EXHIBIT A

NF-461/03 NCL-65/2003

US, WO

FORM 2

THE PATENTS ACT-1970

COMPLETE SPECIFICATION

(See section 10)

A BIOLOGICAL PROCESS FOR THE SYNTHESIS OF OXIDE NANOPARTICLES

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, Rafi Marg, New Delhi-10001, India, an Indian registered body incorporated under Registration of Societies Act (Act XXI of 1860)

The following specification particularly ascertains the nature of this invention and the manner in which it is to be performed:

Field of the invention

This invention relates to a biological process for the synthesis of oxide nanoparticles by simple exposure of suitable aqueous metal ions to a hydrolyzing fungus. More particularly it relates to a biological process for the synthesis of oxide nanoparticles by the reaction of suitable electrolyte solution with a hydrolyzing wet fungus. Still it relates to a biological process for the synthesis of oxide nanoparticles controlled by the proteins secreted by the respective fungus, which are responsible for the size and shape control of the desired oxide nanoparticles after separating the biomass. More particularly it relates to a method for producing shape, size and polymorph controlled oxide nanoparticles such as titanium oxide (TiO_2), zirconium oxide (ZrO_2), silicon oxide (SiO_2), zinc oxide (ZnO) by using naturally occurring biomaterials such as wet fungus or their extract. The shape, size and polymorph controlled particles formed by this process can be used in numerous technological and medical applications, e.g., electronics, as advanced ceramics, catalysts, sensors, semiconductors, pigments, can be used in cosmetic and medical industries and many others. All publications and patents mentioned in the above specification are herein incorporated by references. While in the foregoing specification, this invention has been described in relation to certain preferred embodiments thereof and many details have been set forth for purpose of illustration, it will be apparent to those skilled in the art that the invention is susceptible to additional embodiments and that certain of the details described herein can be varied considerably without departing from the basic principles of the invention.

Background of the Invention

Biological and materials synthesis and transformation are one of the core industries of world economy. Various techniques have been developed for large-scale generation

of inorganic materials of controllable structure and size, some based on physical and some on chemical principles. Numerous substances are synthesized using processes that require non-ambient temperatures and/or non-ambient pressures that require capital-intensive equipment. Methods that can produce useful chemicals and materials at conditions closer to ambient conditions that use simple equipments are economically, ecologically and environmentally more desirable.

Description of the Prior art

Significant research efforts have been devoted for nanostructure synthesis as a means to achieve materials having commercial requirements in areas as diverse as ceramics, electronics, pigments, cosmetics and medical industries (Mann et al., *Nature.*, 1996, *382*, 313; Matijevic et al., *Curr. Opin. Colloid Interface Sci.*, 1996, *1*, 176). To date, zirconia-based ceramic alloys have been demonstrated to be the strongest and toughest oxide ceramics yet produced. ZrO_2-toughened ceramics, in which the toughening process is based on the ZrO_2 transformation, have been used in many materials systems. Pure ZrO_2 at ambient temperature is polymorphic, exhibiting cubic (C) (fluorite) structure (Fm3m) at higher temperatures (>2370°C), tetragonal structure ($P4_2/nmc$) at intermediate temperatures (1200°-2370°C) and monoclinic symmetry ($P2_1/c$) at low temperatures (<950°C) (Monte *et al.*, *J. Am. Ceram. Soc.* 2000. 83, 628). The most dramatic increase in its industrial applicability has been brought about by the discovery that the t→m transformation on cooling below 950°C can be controlled by suitable material processing to become the source of transformation plasticity and transformation toughening in tailored, two-phase microstructures (Garvie *et al.*, Nature. 1975, 258, 703). Transformation toughening of ZrO_2 was first reported in a paper entitled "Ceramic Steel" (Garvie *et al.*, *Nature* 1975, 258, 703). Kisi and Howard (1998 in Key Engineering Materials. Vols 153-154, pp1-36) provide

a recent review if the phases observed in ZrO_2 and its alloys. Various efforts have been made in the fabrication of highly crystalline gallium oxide nano tubes, wires and brushes using molten gallium and microwave plasma treatment (Sharma et al, *J. Am. Chem. Soc.*, 2002, 124, 12288), hierarchical ZnO nanostructures by vapor transport and condensation technique (Lao *et al*, *Nano Lett.*, 2002, 2, 1287).

Stober et. al have synthesized silica nanoparticles in the size range of 200-2000 nm. Silica nanospheres were produced by reaction between tetraesters of silicic acid (tetraalkyl silicates) with ammonia as a catalyst in presence of alcohol. (Stober, Fink and Bohn, *J. Colloid Interface Sci.* 1968, 26, 62). New structures (hexagons) of amorphous silica were synthesized *in vitro* by incubating 50 mg/ml poly-L-lysine (PLL) in 1M pre-hydrolysed tetramethoxysilane (Patwardhan et al, *Chem.Commun.* 2003, 1122).

Titanium, zirconium and hafnium dioxides were synthesized by using self-assembled monolayers of organosilicon hydrides ($RSiH_3$). The reactions of alkyl-, fluoroalkyl-, and w-alkenyl-silanes and alpha-w-bis-hydridosilanes with nanoporous TiO_2 (anatase), ZrO_2 (monoclinic) and HfO_2 (monoclinic) powders were synthesized (Fadeev *et al Langmuir* 2002, 18, 7521). Lin Shi *et al* have synthesized nanosized TiO_2 in the pore channels of a silica-based mesoporous thin films (Lin Shi *et al Advanced Materials* 2002, 14, 830). Amorphous titanium dioxides were coated with colloidal polystyrene spheres. The core-shell particles can be turned into spherical hollow titania shells by dissolution of the polystyrene cores in suspension or by calcination of the dried particles in a furnace (Imhof *et al Langmuir* 2001, *17*, 3579).It is known that the presence of impurity in semiconducting nanoparticles results in increasing photodegradation rate and impurities plays important roles in modulating the properties of semiconducting nanoparticles, which leads to the concept of

introduction of dopant to optimize the semiconducting behavior or new crystalline phase. Film nanostructure has been optimized for maximum photodegradation efficiency by controlling the original reverse micellar composition, the ripening of the particles, and the thickness of the films. Films doped with silver ions, incorporated through the reverse micellar route, are more efficient photocatalysts than pure titanium films and become even more efficient when they are treated with UV irradiation. Films doped with ruthenium ions are less efficient for photocatalysis but when they are treated with UV radiation, they also become more efficient photocatalysts than pure titania films (Stathatos *et al Langmuir* 2001, 17, 5025). Iron-doped titania photocatalysts with different iron contents were prepared by using a sol-gel method in acidic media (Wang *et al J. Phys. Chem. B* 2001, 105, 9692). Braun et al has reported the stable suspension of fluorescent erbium-doped titania nanoparticles and their assembly into thin films and photonic crystals (Braun *et al Chem. Mater.* 2003, 15, 1256). Crystalline TiO_2 films have been deposited on several substrates (glass, F-doped SnO_2-covered glass, and silicon wafers) by a drain-coating method from a colloidal anatase aqueous solution at low temperature (Peiro *et al. Chem. Mater.* 2001, 13, 2567). Butanediol sol-gel synthesis for Fe doped titania which allows to control the material properties on atomic, mesoscopic, and macroscopic scales with respect to structure-morphology, property and relationships (Zhang *et al. Chem. Mater.* 2003, 15, 4028). Zirconia nanoparticles doped with up to 50 mol % Al_2O_3 were prepared by chemical vapor synthesis (CVS) (Srdic *et al. Chem. Mater.* 2003, 15, 2668). Ultrasound radiation was used to prepare Eu_2O_3 in zirconia and yttrium-stabilized zirconium (YSZ) nanoparticles. (Gedanken *et al J. Phys. Chem. B* 2000, 104, 7057). Erbium-doped ZrO_2 nanoparticles are prepared by a sol-emulsion-gel technique. The effects of the Er^{3+} concentration and different codopants (Yb^{3+} and

Y^{3}) in the ZrO^{2} matrix on the up converted emission are observed. (Prasad *et al. J. Phys.Chem. B* 2002, 106, 1909).

US Pat. No. 6,136,186 used a method and apparatus for mineralizing organic contaminants in water or air provides photochemical oxidation in a two-phase or three-phase boundary system formed in the pores of a TiO_2 membrane in a photocatalytic reactor. In the three-phase system, gaseous (liquid) oxidant, liquid (gaseous) contaminant, and solid semiconductor photocatalyst meet and engage in an efficient oxidation reaction. The porous membrane has pores, which have a region wherein the meniscus of the liquid varies from the molecular diameter of water to that of a capillary tube resulting in a diffusion layer that is several orders of magnitude smaller than the closest known reactors. The photocatalytic reactor operates effectively at ambient temperature and low pressures.

US Pat. No. 6,586,095 used a method for preparing a plurality of semiconductor oxide nanostructures that have a substantially rectangular cross-section from an oxide powder is disclosed. A representative method includes: heating the oxide powder to an evaporation temperature of the oxide powder for about 1 hour to about 3 hours at about 200 torr to about 400 torr in an atmosphere comprising argon; evaporating the oxide powder; and forming the plurality of semiconductor oxide nanostructures.

The prior art methods for the growth of various oxide nanoparticles teach us to grow a wide variety of these particles together with the control over their crystal size, shape and morphology but have certain limitations.

The major drawbacks of the prior art processes compare to the present invention are:

1. The methods are not environmentally friendly and simple.
2. Large-scale synthesis is not possible
3. Not Cost effective/Economical system for the industry

4. Uniform size control is tough
5. Complex experimental conditions
6. Require more manoeuvring
7. Not a robust system
8. Stability of the system is low
9. There is an upper limit to scaling in terms of mass production
10. Possibility of contamination is high if proper care is not taken.

To reduce the complexity of the above mentioned processes and to enhance the large-scale production of these oxide nanoparticles, a very efficient and simple biological method has been invented which was realized by challenging micro-organism for the synthesis of semiconducting oxide nanoparticles by simple exposure of suitable metal salts to various hydrolysing fungi or their extract which resulted in controlled size, shape and polymorphs of the desired oxide nanoparticles which are environmental friendly thereof.

Some examples are given in the table below.

Fungus	Metal salt	Oxide type	Polymorph	Shape	Size
Fusarium Oxysporum	K_2TiF_6	TiO_2	Brookite	spherical/ square	10 nm to 50 nm
Fusarium oxysporum	K_2ZrF_6	ZrO_2	Baddelyte (Monoclinic)	Spherical	2 nm to 20 nm
Fusarium oxysporum	K_2SiF_6	SiO_2	Silica (crystalline/amorphous)	spherical/ circular	20 nm to 200 nm
Fusarium sp.	K_2TiF_6	TiO_2	Mixture (Brookite/ Rutile)	Spherical/ square	20 nm to 50 nm
Fusarium sp.	K_2ZrF_6	ZrO_2	Baddelyte (Monoclinic)	Spherical	2 nm to 20 nm
Fusarium sp.	K_2SiF_6	SiO_2	Silica (crystalline/amorphous)	spherical/circular	20 nm to 200 nm
Trichothecium sp.	K_2TiF_6	TiO_2	Brookite	spherical/ square	20 nm to 50 nm

Objects of the invention

The main object of the invention is to provide a biological process for the synthesis of semiconducting oxide nanoparticles, which are environmental friendly.

It is another object of the invention to provide a process for the preparation of shape, size and polymorph controlled synthesis of oxide nanoparticles that are user friendly.

It is yet another object of the invention to provide an economic and efficient process for the synthesis of shape, size and polymorph controlled oxide nanoparticles.

These and other objects of the invention are achieved by the process of the invention, which uses a biological method for the synthesis of shape, size and polymorph controlled oxide nanoparticles.

Summary of the invention

Accordingly the present invention provides a biological process for the synthesis of shape, size and polymorph controlled oxide nanoparticles, which comprises incubation of wet fungus or their extract with an aqueous solution of metal salt of which the oxide particle are to be prepared at a temperature in the range of 15 to 40 °C for 1 to 3 days, by separating the biomass through a filter of minimum 1 micron pore size to obtain the oxide nanoparticles.

In one of the embodiments of the present invention the metal salts may be from transition metal group.

In another embodiment, the metal salts may be of chloride, nitrares, oxalates or sulfates.

In yet another embodiment the concentration of the salt of metal ions may be a minimum of 1mM.

In yet another embodiment of fungus biomass used may be 10 to 60 grams.

In still another embodiment the fungi used are genera of *Fusarium* sp., *Trichothecium*

sp.. *Verticillium* sp., *Chloridium* sp., *Aspergillus* sp., *Cephaliophora* sp., *Fusarium oxysporum*, *Helicostylum* sp.

In another embodiment the fungus is used in the form of a whole cell, wet solid mass or their extract.

In yet another embodiment of the invention, the temperature for incubation is in the range of 23-33 °C., preferably 25-29 ^{0}C.

The process of the invention is described herein below with reference to the following examples, which are illustrative and should not be construed to limit the scope of the invention, in any manner.

EXAMPLE 1

This example illustrates the synthesis of titanium oxide (TiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium oxysporum*) which was maintained on potato-dextrose-agar (PDA) slants. Stock culture was maintained by sub culturing at monthly intervals. After growing at pH 7 and 27 °C for four days the slants were preserved at 15 °C. From an actively growing stock culture, subculture was made on fresh slant and after four days of incubation at pH 7 and 27 °C was used as the starting materials for fermentation experiment. The fungus was grown in 500 ml Erlenmeyer flask containing 100 ml malt extract-glucose-yeast extract-peptone (MGYP) medium which is composed of malt extract (0.3 %), glucose (1 %), yeast extract (0.3 %) and peptone (0.5 %). After adjusting the pH of the medium to 7, the cultures was grown with continuous shaking on a rotary shaker (200 rpm) at 27 °C for 96 hours. After 96 hours of fermentation, mycelia were separated from the culture broth by centrifugation (5000 rpm) at 20 °C for 20 minutes and then the mycelia were washed thrice with sterile distilled water under sterile conditions. The harvested mycelia mass (20 g wet

wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M K_2TiF_6 solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Scanning electron microscopy (SEM) and Energy dispersive analysis of X-rays (EDAX). The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form (crystalline phase) and were further characterized.

EXAMPLE 2

This example illustrates the synthesis of titanium oxide (TiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium oxysporum*). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M titanium oxalate [$Ti(C_2O_4)_2$] solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form (crystalline phase) and were further characterized.

EXAMPLE 3

This example illustrates the synthesis of zirconium oxide (ZrO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium oxysporum*). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M K_2ZrF_6 solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form (crystalline phase) and were further characterized.

EXAMPLE 4

This example illustrates the synthesis of zirconium oxide (ZrO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium oxysporum*). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M zirconium oxychloride ($ZrOCl_2$) solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into

other polymorphic form (crystalline phase) and were further characterized.

EXAMPLE 5

This example illustrates the synthesis of silicon oxide (SiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium oxysporum*). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M K_2SiF_6 solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form (crystalline phase) and were further characterized.

EXAMPLE 6

This example illustrates the synthesis of silica (SiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium* sp.). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M tetraethyl ortho silicate (TEOS) solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was

calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form and were further characterized.

EXAMPLE 7

This example illustrates the synthesis of silica (SiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium* sp.). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M tetramethyl ortho silicate (TMOS) solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form and were further characterized.

EXAMPLE 8

This example illustrates the synthesis of silica (SiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium* sp.). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in 100 ml of 10^{-3} M silicic acid (H_2SiO_3) solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The

nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form and were further characterized.

EXAMPLE 9

This example illustrates the synthesis of silica (SiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium* sp.). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in a 100 ml of mixture of 10^{-3} M silicic acid (H_2SiO_3, 50 ml) and of 10^{-3} M (50 ml) tetraethyl ortho silicate (TEOS) solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form and were further characterized.

EXAMPLE 10

This example illustrates the synthesis of titanium dioxide (TiO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium* sp.). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in a 100 ml mixture of 10^{-3} M K_2TiF_6 (50 ml) and 10^{-3} M (50 ml) of titanium oxalate [$Ti(C_2O_4)_2$] solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract

time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form and were further characterized.

EXAMPLE 11

This example illustrates the synthesis of zirconium dioxide (ZrO_2) nanoparticles by using a hydrolyzing fungus, (*Fusarium* sp.). (Fungus culturing details - see Example 1). The harvested mycelia mass (20 g wet wt. of mycelia) was then resuspended in a 100 ml mixture of 10^{-3} M K_2ZrF_6 (50 ml) and 10^{-3} M (50 ml) of zirconium oxychloride ($ZrOCl_2$) solution in 500 ml Erlenmeyer flasks. The whole mixture was put in to a shaker at 27 °C (200 rpm) and the reaction carried out for a period of 48 hours. The bio transformation were collected by separating the fungal mycelia or aqueous extract time to time by filtration from the solution containing the mycelia inside the inoculation chamber under stirrer condition and was monitored by periodic sampling of aliquots (10 ml) of the aqueous solution and was characterized. The nanoparticles formed were dried and the powder formed was calcined at 100, 300, 600 and 800 °C for the phase transformation into other polymorphic form and were further characterized.

Advantages of the process claimed in the present invention are:

1. The main advantage of the present invention is the use of biological process for the synthesis of shape, size and polymorph controlled oxide nanoparticles, which comprises incubation of wet fungus or their extract with an aqueous solution of metal salt of which the oxide particle are to be prepared at a temperature in the range of 15 to 40 °C for 1 to 3 days, by separating the biomass through a filter of minimum 1 micron pore size to obtain the oxide nanoparticles.
2. The main advantage of the present invention is the use of naturally occurring fungi under aqueous medium.
3. Another major advantage of the present invention is that the oxide nanoparticles formed are quite stable in the aqueous solution.
4. Another advantage of the present invention is that the oxide nanoparticles formed are of highly controlled in shape.
5. Another major advantage of the present invention is that different polymorphism (crystalline phase) of the suitable oxide nanoparticles can be achieved by using suitable fungal mass.
6. Uniform size control
7. Large scale synthesis is possible
8. Ambient experimental conditions
9. Cost effective/Economical system for the industry
10. The method of the invention is also environmentally friendly and simple.
11. Another advantage of the present invention is that the use of various metal salts of chloride, nitrates, oxalates or sulfates.
12. Another advantage of the present invention is use of naturally occurring

fungus in the form of a whole cell, wet solid mass or their extract.

13. Another advantage of the present invention is the temperature for incubation is in the range of 23-33 °C., preferably 25-29 ^{0}C.

We claim:

1. A biological process for the synthesis of shape, size and polymorph controlled oxide nanoparticles, which comprises incubation of wet fungus or their extract with an aqueous solution of metal salt of which the oxide particle are to be prepared at a temperature in the range of 15 to 40 °C for 1 to 3 days, by separating the biomass through a filter of minimum 1 micron pore size to obtain the oxide nanoparticles.
2. A process as claimed in claim 1 wherein naturally occurring fungi were used under aqueous medium.
3. A process as claimed in claim 1 wherein the oxide nanoparticles formed are quite stable in the aqueous solution.
4. A process as claimed in claim 1 wherein the oxide nanoparticles formed are of highly controlled in shape.
5. A process as claimed in claim 1 wherein different polymorphism (crystalline phase) of the suitable oxide nanoparticles can be achieved by using suitable fungal mass.
6. A process as claimed in claim 1 wherein uniform size control is possible.
7. A process as claimed in claim 1 wherein large-scale synthesis of oxide nanoparticle is possible.
8. A process as claimed in claim 1 which uses ambient experimental conditions.
9. A process as claimed in claim 1 which is cost effective/economical system for the industry.
10. A process as claimed in claim 1 which is a environmentally friendly and simple process.

11. A process as claimed in claim 1, which uses various metal salts of chloride, nitrates, oxalates or sulfates.
12. A process as claimed in claim 1, which is uses naturally occurring fungus in the form of a whole cell, wet solid mass or their extract.
13. A process as claimed in claim 1, which is the temperature for incubation is in the range of 23-33 °C., preferably 25-29 ^{0}C.
14. A process as claimed in claim 1 wherein the concentration of the salt of metals may be a minimum of 1mM.
15. A process as claimed in claim 1 wherein the fungus extract used may be 10 to 60 mgs.
16. A process as claimed in claim 1 wherein the fungi used are genera of *Fusarium* sp., *Trichothecium* sp., *Verticillium* sp., *Chloridium* sp., *Aspergillus* sp., *Cephaliophora* sp., *Fusarium oxysporum*, *Helicostylum* sp.

Dated this _____ day of June 2002

Scientist
IPMD
COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH
NewDelhi-110 067

Abstract

This invention relates to a biological process for the synthesis of size, shape and polymorph controlled semiconducting oxide nanoparticles by simple exposure of suitable aqueous metal ions to a hydrolyzing fungus or their extract. The oxide nanoparticles were achieved by the reaction of suitable electrolyte solution with the hydrolyzing wet fungus. The size, shape and the polymorph selectivity in the synthesis of desired oxide nanoparticles is controlled by the proteins secreted by the respective fungus during their growth. More particularly it relates to a method for producing size, shape and polymorph controlled oxide nanoparticles such as titanium oxide (TiO_2), zirconium oxide (ZrO_2), silicon oxide (SiO_2), zinc oxide (ZnO) by using naturally occurring bio-materials such as wet fungus or their extract.